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# TECHNICAL NOTE

D-1094

EFFECT OF STRAIN RATE ON MECHANICAL PROPERTIES

OF WROUGHT SINTERED TUNGSTEN AT

TEMPERATURES ABOVE 2500° F

By Paul F. Sikora and Robert W. Hall

Lewis Research Center Cleveland, Ohio

NATIONAL AERONAUTICS AND SPACE ADMINISTRATION
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# NATIONAL AERONAUTICS AND SPACE ADMINISTRATION

## TECHNICAL NOTE D-1094

EFFECT OF STRAIN RATE ON MECHANICAL PROPERTIES OF WROUGHT

SINTERED TUNGSTEN AT TEMPERATURES ABOVE 2500° F

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#### SUMMARY

Specimens of wrought sintered commercially pure tungsten were made from 1/8-inch swaged rods. All the specimens were recrystallized at  $4050^{\circ}$  F for 1 hour prior to testing at temperatures from  $2500^{\circ}$  to  $4000^{\circ}$  F at various strain rates from 0.002 to 20 inches per inch per minute.

Results showed that, at a constant temperature, increasing the strain rate increased the ultimate tensile strength significantly. The effects of both strain rate and temperature on the ultimate tensile strength of tungsten may be correlated by the linear parameter method of Manson and Haferd and may be used to predict the ultimate tensile strength at higher temperatures,  $4500^{\circ}$  and  $5000^{\circ}$  F. As previously reported, ductility, as measured by reduction of area in a tensile test, decreases with increasing temperature above about  $3000^{\circ}$  F. Increasing the strain rate at temperatures above  $3000^{\circ}$  F increases the ductility. Fractures are generally transgranular at the higher strain rates and intergranular at the lower strain rates.

#### INTRODUCTION

As part of a broad program of research on tungsten and its alloys at the NASA Lewis Research Center, the mechanical properties of these materials at higher temperatures are under investigation. Results of high-temperature tensile tests of wrought sintered tungsten at temperatures of 2500° to 4400° F have been reported previously (refs. 1 and 2). In the course of the previous work, it was noted that the strength of tungsten at high temperatures is unusually strain-rate-dependent. Since some of the potential applications of tungsten at high temperatures involve high rates of loading and since little information on the mechanical properties of tungsten at high strain rates and high temperature is available in the literature, this investigation was undertaken to determine the effect of strain rate on the tensile strength and ductility of tungsten at high temperatures. This report describes the results of

tensile tests conducted at temperatures from 2500° to 5000° F at strain rates from approximately 0.002 to 20 inches per inch per minute.

#### MATERIALS, APPARATUS, AND PROCEDURE

#### Materials

The material used in this investigation was commercially pure sintered tungsten bar stock swaged to 1/8-incl diameter. A chemical analysis of the as-swaged bars is shown in the following table:

| Element                        | Fe | Мо  | Cr | Si | С  | 0 | N |
|--------------------------------|----|-----|----|----|----|---|---|
| Composition, parts per million | 20 | 150 | \$ | <3 | 49 | 6 | 1 |

Tensile specimens of the type shown in figure 1 were ground from the 1/8-inch-diameter bars and were then subjected to a recrystallization treatment of  $4050^{\circ}$  F for 1 hour in a vacuum of 0.1 micron. This treatment at a temperature slightly higher than the highest test temperature initially planned ( $4000^{\circ}$  F) was selected to furnish a uniform material that would remain relatively unchanged over the entire test range during heating and testing. Photomicrographs of the as-swaged and recrystallized material are shown in figure 2. Grain size of the recrystallized material was 6 to 8.

#### Apparatus

Tensile testing machine. - Two commercial, screw-driven, tensile testing machines were used in this investigation. One machine had a crosshead speed that was continuously variable from 0.03 to 20 inches per minute by means of a variable-speed motor equipped with a thyratron control. The other machine had a crosshead speed that could be operated at either a fixed speed or continuously variable up to the fixed speed. The fixed speed could be changed by a factor of 10 by changing a shift lever or could be changed to another speed by changing two gears in the control train. On both machines, load agains: time curves were autographically recorded, using strain-gage-type load cells to measure load.

<u>Vacuum test chamber</u>. - The test chamber is a water-cooled, stainless-steel vacuum chamber that is split longitudinally and hinged at one side as shown in figure 3. Iubricated "O" rings at the top and bottom of the chamber provide sliding seals between the pull rods and the chamber. The vacuum system is described in reference 1.

Heater assembly and power supply. - Details of the heater assembly are shown in figure 4. The heater consists of a 5/8-inch-diameter by 5-inch-long seamless tantalum tube slit to within 1/2 inch of the bottom and welded to heavier tantalum sheet connectors. This arrangement in which the heating tube is gripped only at the top allows it to expand and contract freely on heating and cooling. Tantalum reflectors surround the heater.

The power supply consists of a 22 KVA transformer controlled by a variable autotransformer. Output on the secondary of the transformer is 10 volts at 2200 amps and is fed to the heater through water-cooled electrodes in the chamber.

Tensile specimen, grips, and loading fixtures. - A pin-type concept of gripping was employed in these tests wherein the cylindrical specimen is pinned in the grips as shown in figure 4. The specimen is gripped outside the heater assembly. The thermal gradient along the 1-inch gage length was  $\sim 35^{\circ}$  at  $4000^{\circ}$  F. The loading fixtures are described in reference 1.

#### Procedure

Temperature measurement and control. - Temperatures up to  $4000^{\circ}$  F were measured by means of W-Mo thermocouples spotwelded to the surface of the specimen at the midpoint of the gage length. Tests at  $4500^{\circ}$  to  $5000^{\circ}$  F utilized W-W26 Re thermocouples. Control of the temperature was accomplished by manually adjusting the variable autotransformer to the desired power level.

Test procedure. - All tests were conducted in a vacuum at a pressure of less than 0.1 micron. The specimen was heated to the test temperature in about 25 minutes and held at temperature about 10 minutes. It was then loaded to fracture at a constant crosshead speed of 0.002, 0.2, or 20 inches per minute. Some specimens were loaded to about 0.5percent deformation at (1) a crosshead speed of 0.002 inch per minute and then to fracture at a crosshead speed of 0.02 inch per minute or (2) a crosshead speed of 0.2 inch per minute and then to fracture at a crosshead speed of 2 inches per minute. The data from this step-type procedure are plotted at the higher strain rate since the strain at the 0.5-percent deformation is so small relative to the total strain that no changes were observed in either tensile strength or ductility, and the results were equivalent to those conducted at a constant crosshead speed. Crosshead speed was assumed to be the strain rate in the gage length on the basis of a uniform diameter and thermal gradient within the 1-inch gage length. Actual strain rates are lower than the nominal rates in the elastic region but approximate the crosshead speed in the plastic region.

Total elongation measurements were made by fitting the fractured halves together and measuring between the shoulders of the reduced section. Percent elongation values were calculated (table I) assuming an effective gage length of 1 inch.

#### RESULTS AND DISCUSSION

Effect of Strain Rate on Ultimate Tensile Strength

at Temperatures From 2500° to 4000° F

The results of the tensile tests at temperatures up to 4000° F are plotted as a function of strain rate for the various test temperatures in figure 5 and are tabulated in table I. There is an appreciable increase in ultimate tensile strength with increasing strain rate at all test temperatures. For example, the ultimate tensile strength of recrystallized tungsten at 2500° F increases from 15,000 to 30,000 psi with an increase in strain rate from 0.002 to 20 inches per inch per minute. Similarly, at 4000° F the ultimate tensile strength is increased from 3200 to 13,000 psi for the same increase in strain rate. Equally large increases in 0.2-percent offset yield strength, as determined from the load against time curves, resulted from increased strain rate (table I). Unfortunately the 0.2-percent offset yield strength data showed considerable scatter, probably as a result of errors resulting from the use of crosshead motion as a measure of elongation in the elastic range.

From the data it is apparent that, for applications involving rapid loading rates, considerably more strength is available than the results of conventional short-time tensile tests would suggest. When translating this effect into possible operating temperature, it may be seen that for a maximum design fracture stress of 10,000 psi the temperature limit is increased from  $3000^{\circ}$  to  $4000^{\circ}$  F as the strain rate is increased from 0.02 to 2 inches per inch per minute.

In figure 6 the ultimate tensile strength (ata are plotted as a function of temperature at the various strain rates. This plot indicates that at all temperatures strength increases substantially with increasing strain rate.

Correlation of Ultimate Tensile Strength, Strain Rate,

and Temperature by Linear Parameter Method

The similarity of short-time tensile strength data at various strain rates and temperatures to stress-rupture data suggested that

parameter methods of the type used for correlating and extrapolating short-time rupture data might be applicable to these data. By using the method of least squares described in reference 3 to optimize the parametric constants in the Manson-Haferd linear parameter (ref. 4) it was found that the data could best be represented by the equation  $\sigma = f(P)$  where

$$-P = \frac{T - 1600}{\log y - 4.8687}$$

and

σ ultimate tensile strength, psi

T temperature, OF

y reciprocal of strain rate

Using this parameter, a master curve of stress against -P was constructed (fig. 7) using all the data for the five different strain rates and temperatures from  $2500^{\circ}$  to  $4000^{\circ}$  F. From this master curve, the optimized curves shown in figure 8 were plotted showing the ultimate tensile strength as a function of the strain rate for the various test temperatures. Actual data points are also plotted to show the fit of the calculated curve to the data. Except at the lowest temperature,  $2500^{\circ}$  F, the calculated curves fit the experimental data very well.

One of the prime reasons for treating the data by the linear parameter method is to enable extrapolation of the data to higher temperatures where testing is extremely difficult. In order to determine the utility of the method for predicting the ultimate tensile strength of tungsten as a function of strain rate at temperatures above 4000° F, the master curve was used to predict the variation of ultimate strength with strain rate at 4500° and 5000° F. The calculated curves shown in figure 9 are the results of additional tensile tests conducted at 4500° and 5000° F at strain rates of 0.2 and 20 inches per inch per minute. The predicted and experimental data are also tabulated in table II. There is good agreement between the predicted values and the actual values at both temperatures and strain rates. For example, the predicted value at 4500° F and a strain rate of 20 inches per inch per minute was 10,200 psi, and the actual values obtained were 10,300 and 9900 psi; at 5000° F and 20 inches per inch per minute the predicted value was 7000 psi, and the actual was 6700 psi; at 4500° F and 0.2 inch per inch per minute the predicted value was 4600 psi, and the actual strength was 4700 psi. The agreement of the predicted ultimate tensile strength (3700 psi) and the actual strength (2800 psi) at 5000° F and a strain rate of 0.2 inch per inch per minute was not as good as the previous examples, but was reasonably close. An examination of the fractured edge of this specimen shows a typical single crystal type of fracture, chisel edge, indicating that

considerable grain growth occurred during the test. This structural instability probably accounts for the poorer agreement of prediction and experiment and suggests caution in applying the parameter method for extrapolation into regions where large structure changes are to be expected.

It is apparent then that the ultimate tensile strength of tungsten at temperatures of  $4500^{\circ}$  and  $5000^{\circ}$  F may be predicted with a fair degree of accuracy, based on tests conducted at lower temperatures. This may be an important consideration since it is much more difficult to attain temperatures in excess of  $4000^{\circ}$  F than temperatures below  $4000^{\circ}$  F.

## Effect of Strain Rate on Ductility of

#### Tungsten at High Temperatures

In previous investigations of the high-temperature mechanical properties of wrought sintered tungsten, it was noted that the ductility, as measured by the reduction of area in a tensile test, decreases appreciably with increasing temperature above about 2500° to 3000° F. The effect of strain rate on high-temperature ductility (again using the reduction of area as a criterion) is shown in figure 10. At 2500 F the ductility is high (99-percent reduction area) for all strain rates; at 3000° F the ductility remains high (99 percent) at the highest strain rates (0.2 in./in./min and above) and decreases considerably (22 percent at a strain rate of 0.002 in./in./min) at the lower strain rates. At 3500° and 4000° F the ductility is high (99 percent) only at the highest strain rate, 20 inches per inch per minute, and decreases rapidly with decreasing strain rate. It was also noted that the ductility remained high (99 percent) at all temperatures from  $2500^{\circ}$  to  $5000^{\circ}$  F at a strain rate of 20 inches per inch per minute. The minimum ductility occurs at temperatures of 3500° to 4000° F and increases as the strain rate increases from 0.002 to 20 inches per inch per minute. It may be seen that the ductility increases with increasing temperature above 4000° F at a strain rate of 0.2 inch per inch per minute. Elongation showed similar trends but with considerably more erratic behavior (table I).

Microstructural examination of the fractured specimens indicated that the decrease in ductility with increasing temperature is associated with a change in the mode of fracture from transgranular at the lower temperatures to intergranular at the higher temperatures. This is illustrated in figure 11 by comparing photomicrographs of specimens fractured at 2500° and 4000° F at a strain rate of 0.002 inch per inch per minute. At 2500° F, fracture was predominantly transgranular and ductility was high (99-percent reduction of area). The major effect of increasing strain rate was to increase the temperature at which intergranular fracture occurred. At the highest strain rate, 20 inches per inch per minute, fracture remained transgranular up to the highest temperature, 5000° F, and ductility was high at all temperatures.

Figure 12 compares the fractures of specimens evaluated at  $3000^{\circ}$  F at rates of 20, 2, 0.2, 0.02, and 0.002 inch per inch per minute. An increase in intergranular cracking at the lower strain rates may be seen in the photomicrographs.

#### SUMMARY OF RESULTS

On the basis of the these results, the following summarization of the mechanical properties of recrystallized, wrought, sintered tungsten in the  $2500^{\circ}$  to  $5000^{\circ}$  F range may be made:

- l. At a constant temperature, increasing the strain rate increases the ultimate strength significantly. For example, increasing the strain rate from 0.002 to 20 inches per inch per minute doubles the strength at  $2500^{\circ}$  F (from 15,000 to 30,000 psi) and approximately quadruples it at  $4000^{\circ}$  F (from 3200 to 13,500 psi).
- 2. The effects of both temperature and strain rate on the ultimate tensile strength of tungsten may be correlated by the linear parameter (Manson-Haferd method)

$$-P = \frac{T - 1600}{\log y - 4.8687}$$

where

- T temperature, <sup>O</sup>F
- y reciprocal of strain rate

Use of this parameter to predict tensile strength at  $4500^{\circ}$  and  $5000^{\circ}$  F from  $2500^{\circ}$  to  $4000^{\circ}$  F data proved to be successful.

3. As previously reported, the ductility of tungsten (as measured by reduction of area in a tensile test) decreases with increasing temperature above about  $3000^{\circ}$  F. The effect of increasing the strain rate at temperatures above  $3000^{\circ}$  F is to increase the ductility. Fractures are generally transgranular at the higher strain rates and intergranular at the lower strain rates.

Lewis Research Center
National Aeronautics and Space Administration
Cleveland, Ohio, August 9, 1961

#### REFERENCES

- 1. Hall, Robert W., and Sikora, Paul F.: Tensile Properties of Molybdenum and Tungsten from 2500° to 3700° F. NASA MEMO 3-9-59E, 1959.
- 2. Sikora, Paul F., and Hall, Robert W.: High-Temperature Tensile Properties of Wrought Sintered Tungsten. NASA TN D-79, 1959.
- 3. Manson, S. S., and Mendelson, A.: Optimization of Parametric Constants for Creep-Rupture Data by Means of Least Squares. NASA MEMO 3-10-59E, 1959.
- 4. Manson, S. S., and Haferd, Angela M.: A Linear Time-Temperature Relation for Extrapolation of Creep and Stress-Rupture Data. NACA TN 2890, 1953.

TABLE I. - SHORT-TIME TENSILE PROPERTIES OF SWAGED TUNGSTEN AT STRAIN RATES OF 0.002 TO 20 INCHES PER INCH PER MINUTE

E-1375

| Reduction of area, percent       | /min <sup>a</sup> | 66     | 66       | 58    | 45     | 27    | r     | 1.4<br>7.4 | 54           |                       | 66     | 66     | 66     |        | 68     |        | 65     |        |               |        |                  |            |         |        |        |        |        |
|----------------------------------|-------------------|--------|----------|-------|--------|-------|-------|------------|--------------|-----------------------|--------|--------|--------|--------|--------|--------|--------|--------|---------------|--------|------------------|------------|---------|--------|--------|--------|--------|
| Percent<br>elonga-<br>tion       | in./in./m         | 47     | 45       | 48    | 44     | 56    | L     | <u>م</u> 3 | 77           | /in./min <sup>a</sup> | 44     | 48     | 52     |        | 64     |        | 84     |        |               |        |                  |            |         |        |        |        |        |
| 0.2-Percent<br>yield<br>strength | 0.002/0.02        | 2000   | 2500     |       | 3500   | 700   | ()    | 1,000      | T400         | 0.2/2 in.             | 5800   | 0009   | 4300   |        | 4200   |        | 2800   |        |               |        |                  |            |         |        |        |        |        |
| Ultimate<br>tensile<br>strength  | Strain rate,      | 18,500 | 18,200   | 9,300 | 10,700 | 7,000 |       | 4,600      | 4, 800       | Strain rate,          | 30,900 | 28,400 | 22,400 |        | 16,100 |        | 10,500 | :      |               |        |                  |            |         |        |        |        |        |
| Tem-<br>pera-<br>ture            | St                | 2500   |          | 3000  |        | 3500  | (     | 4000       |              |                       | 2500   |        | 3000   |        | 3500   |        | 4000   |        |               |        |                  |            |         |        |        |        |        |
| Reduction of area, percent       |                   | 66     | <u>ග</u> | 22    | 22     | 15    | 14    | 7,7        | <del>-</del> |                       | 66     | 66     | 66     | 66     | 58     | 31     | 37     | 39     |               | 66     | 66               | <b>6</b> 0 | 66      | 66     | 8<br>8 | 66     | 66     |
| Percent<br>elonga-<br>tion       | ./in./min         | 51     | 52       | 22    | 25     | 14    | 12    | CT .       | 74           | in./in./min           | 43     | 40     | 55     | 57     | 56     | 31     | 36     | 36     | /in./min      | 12     | 58               | 47         | 48      | 53     | 58     | 56     | 55     |
| 0.2-Percent<br>yield<br>strength | ate, 0.002 in.    | 4,900  | 5,700    | 1,300 | 1,300  | 1,100 | 2,000 | 1,500      | T, 600       | rate, 0.2 in.,        | 6,700  | 5,600  | 3,800  | 3,800  | 3,800  | 4,800  | 3,500  | 3, 300 | rate, 20 in., | 1 1    | 1<br>1<br>1<br>1 | 9,400      | 8,200   | 10,000 | 10,800 | 7,500  | 8,000  |
| Ultimate<br>tensile<br>strength  | Strain rat        | 14,200 | 16,700   | 5,200 | 5,700  | 4,000 | 5,400 | 5,500      | 5, 100       | Strain re             | 28,400 | 28,600 | 18,400 | 17,600 | 11,800 | 12,500 | 7,000  | 6, 300 | Strain        | 29,400 | 31,800           | 25,700     | 25, 100 | 19,200 | 19,110 | 13,300 | 13,400 |
| Tem-<br>pera-<br>ture            |                   | 2500   |          | 2000  |        | 3500  | (     | 4000       |              |                       | 2500   |        | 3000   |        | 3500   |        | 4000   |        | -             | 2500   |                  | 3000       |         | 3500   |        | 4000   |        |

ATests were run at the lower strain rate to about 0.5-percent deformation and then were increased to the higher strain rate to fracture.

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TABLE II. - EXTRAPOLATED AND ACTUAL ULTIMATE TENSILE STRENGTH

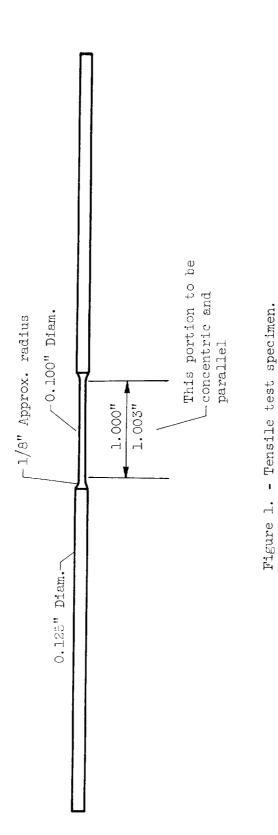
OF SWAGED TUNGSTEN AT 4500° AND 5000° F

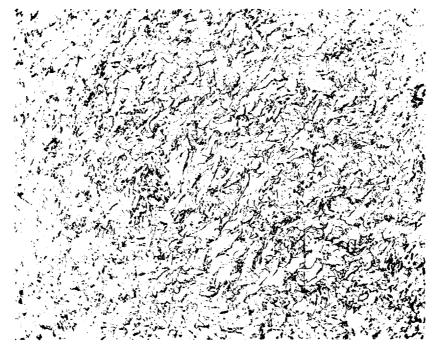
| Tem-<br>pera-<br>ture,<br>OF | Strain rate, in./in./min          | Extrapolated ultimate tensile strength, psi | Actual ultimate tensile strength, psi | Percent<br>elonga-<br>tion | Reduction of area, percent |
|------------------------------|-----------------------------------|---|---------------------------------------|----------------------------|----------------------------|
| 4500                         | 0.002<br>.02<br>.2<br>2.0<br>20.0 | 2,300<br>3,300<br>4,600<br>6,700<br>10,200  | 4,700<br>10,300<br>9,900              | 24<br><br>65<br>33         | 45<br><br>99<br>99         |
| 5000                         | 0.002<br>.02<br>.2<br>2.0<br>20.0 | 1,700<br>3,000<br>3,700<br>4,900<br>7,000   | 2,800                                 | <br>33<br><br>44           | 89<br><br>99               |

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(a) As-swaged.



(b) Recrystallized at  $4050^{\circ}$  F for 1 hour.

Figure 2. - Comparison of as-swaged and recrystallized tungsten. X250. Etchant:
Murakami reagent.

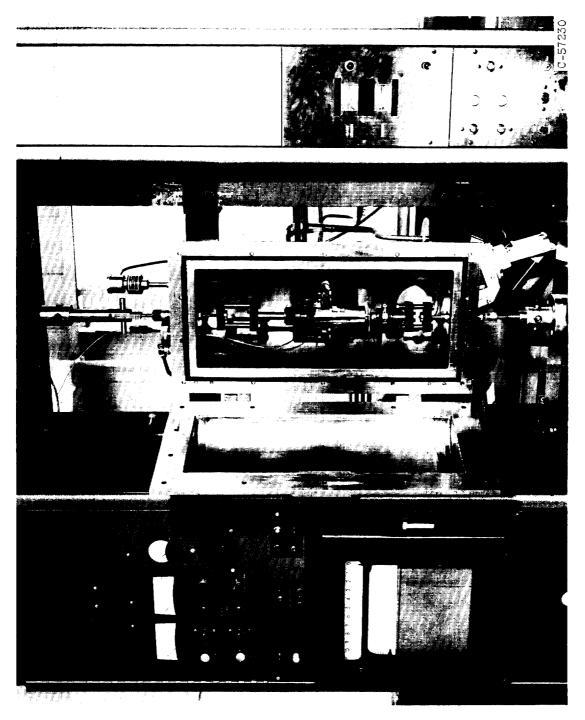
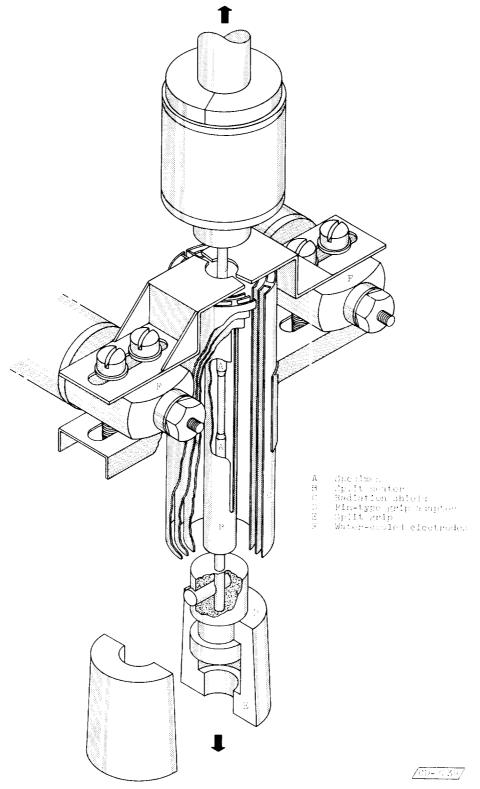
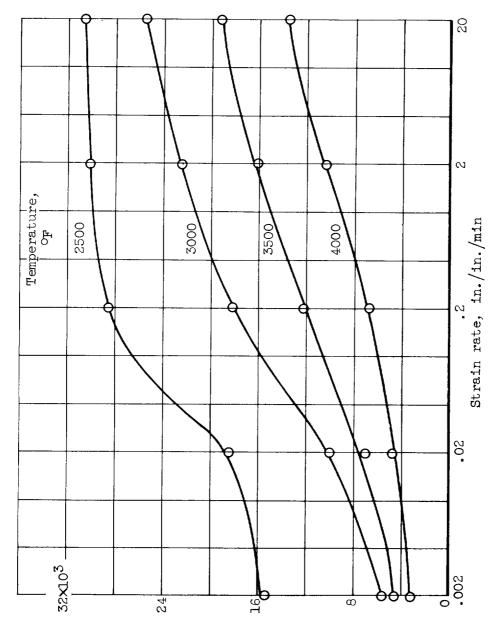


Figure 3. - Vacuum test chamber.

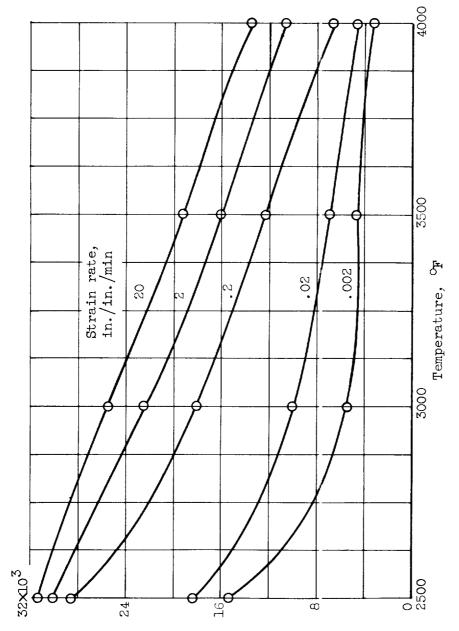


Ff-pure 4. - High-temperature tensile specimen with load train and Leater.



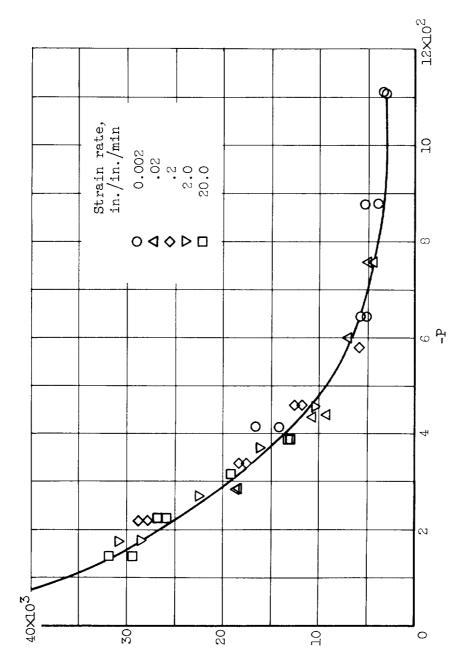
Ultimate tensile strength, psi

Figure 5. - Effect of strain rate on ultimate tensile strength of tungsten at temperatures from  $2500^{\rm O}$  to  $4000^{\rm O}$  F.



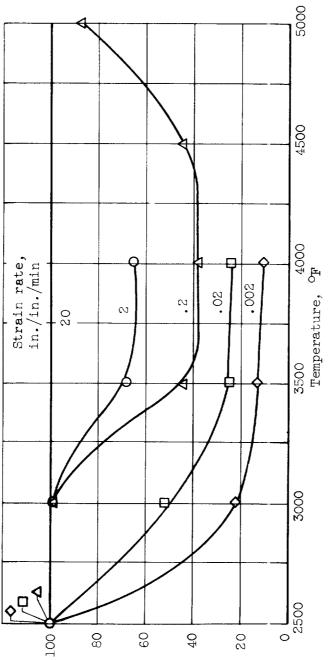
Ultimate tensile strength, psi

Figure 6. - Effect of temperature on ultimate tensile strength of tungsten at various strain rates.



Ultimate tensile strength, psi

Figure 7. - Master curve for ultimate tensile strength plotted against linear parameter.



Reduction of area, percent

Figure 10. - Effect of temperature on ductility of tungsten at various strain rates.

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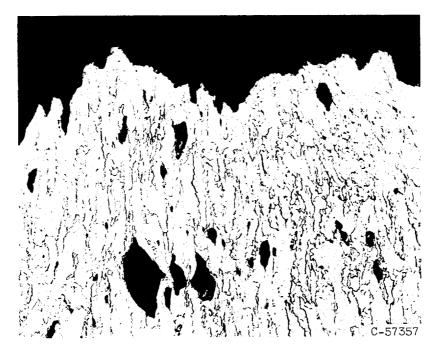
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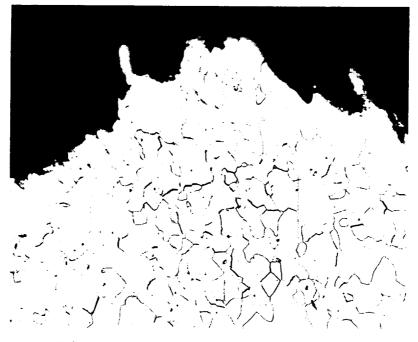


(a) Tested at  $4000^{\circ}$  F and strain rate of 0.002 inch per inch per minute.

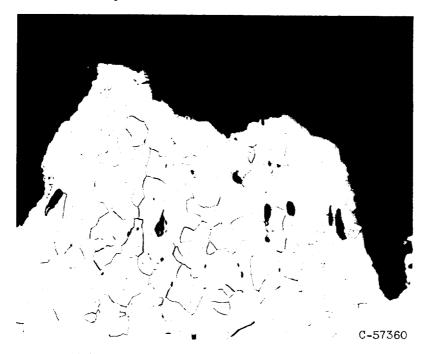


(b) Tested at  $2500^{\circ}$  F and strain rate of 0.002 inch per inch per minute.

Figure 11. - Comparison of mode of fracture with increasing temperature. X250. Etchant: Murakami reagent.

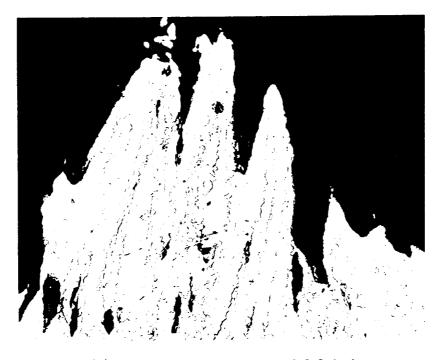


(a) Tested at a strain rate of 20 inches per inch per minute.

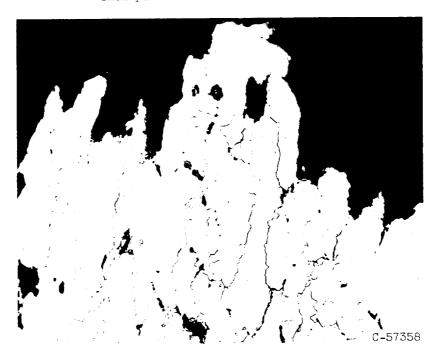


(b) Tested at a strain rate of 2 inches per inch per minute.

Figure 12. - Comparison of mode of fracture for various strain rates at 3000° F. X250. Etchant: Murakami reagent.

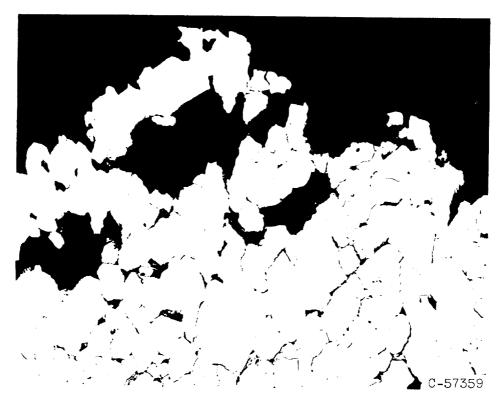


(c) Tested at a strain rate of 0.2 inch per inch per minute.



(d) Tested at a strain rate of 0.02 inch per inch per minute.

Figure 12. - Continued. Comparison of mode of fracture for various strain rates at 3000° F. X250. Etchant: Murakami reagent.



(e) Tested at a strain rate of 0.002 inch per inch per minute.

Figure 12. - Concluded. Comparison of mode of fracture for various strain rates at 3000° F. X250. Etchant: Murakami reageit.